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PUBLISHED BY
CONCRETE PUBLICATIONS LIMITED
14 DARTMOUTH STREET, LONDON, S.W.1

TELEPHONE: WHITEHALL 4581.
TELEGRAPHIC ADDRESS:
CONCRETUS, PARS. LONDON.

PUBLISHERS OF
"CONCRETE & CONSTRUCTIONAL ENGINEERING"
"CONCRETE BUILDING & CONCRETE PRODUCTS"
"CEMENT & LIME MANUFACTURE"
"THE CONCRETE YEAR BOOK"
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VOLUME XXII. NUMBER I

JANUARY, 1949

The Cements Used in the German Motor Highways.

By PROFESSOR DR. HANS KÜHL.

SHORTLY after the occupation of Germany Mr. M. A. Swayze, of the Lone Star Cement Corporation, of the United States, spent three months inspecting the German motor highways (autobahnen), and arrived at the following conclusions¹: "In all my experience I have never seen concrete highways in better shape after at least seven years' service than the German autobahnen. . . . Their quality is a tremendous challenge to American engineers to do likewise." Another inspection was made in the summer of 1947 by a committee of American engineers, whose conclusions are given in a report by Mr. F. H. Jackson and Mr. Harold Allen². After travelling about a thousand miles in the British and American zones, including the "Berlin Ring," the committee found, in some places, damage (cracks, spalling, etc.) similar to that observed on American concrete roads, but in general the opinion of Mr. Swayze was supported with the additional statement that "the quality of the German concrete was excellent." This statement appears to have caused surprise because it was also stated that "German cements are definitely inferior as judged by modern American standards," and "the cements used in the autobahnen were distinctly inferior to those manufactured in this country (the U.S.A.)." The committee added, however, "The concrete is good and from this fact we can conclude at least that it is possible to make good concrete with German cements provided the other conditions are right. . . . Whether the cements were actually inferior remains to be seen." The writer wishes in the following to contribute a little to this interesting problem.

Post-war Difficulties in Germany.

It is obvious, and is admitted by the committee, that the quality of a concrete depends not on the cement only, but also on the aggregate and the workmanship. According to the conclusions of this committee there can be little doubt that the aggregate used was of best quality, that the water-cement ratio was kept as low as possible, that the laying of the concrete with specially-designed machines

represented a high level of technical development, and that all the rules for proper after-treatment of the concrete were adopted. The committee is also of the opinion that the density of traffic and the weights of heavy vehicles were considerably lower than is usual in America, and that the German roads had the benefit of milder winters which obviated the need for melting ice by chlorides. All these points may have contributed to the durability of the concrete, but it is difficult to understand how such excellent concrete could be produced with inferior cement.

One cannot arrive at a valid opinion of German cements, and especially those used in the autobahnen, made before the war by considering the German cement industry after the war. It is agreed that the quality of the cements made in Germany to-day is not the same as those before the war, because the industry is handicapped by various shortages. The degree to which a lost war can react on the quality of cements produced became obvious after 1918. According to the test results published annually by the "Verein Deutscher Portlandzement-fabrikanten" on all the Portland cements made in Germany, the tensile strength dropped by an average of 12 per cent. and the crushing strength by 15 per cent. during the period 1916 to 1919⁸. Considering the far greater damage caused to German industry in the war of 1939 to 1945 it is obvious that the reduction in the quality of cements was also greater.

Mr. Swayze also states that "Their [the German] control of raw-mix proportions was generally lax and fine grinding of these materials which we believe essential for good quality was nowhere near that of American practice. The burning operation is much lighter than ours, even in plants with modern kilns, and free lime contents as high as 2.5 per cent. are not uncommon." These statements are probably quite justified to-day. It is not possible in Germany at present to grind either raw material or cement as finely as is thought necessary. Cement mills are badly worn, and steel cannot be obtained to repair them. In some cement works the mills are lined with boiler plate, and chain-links and similar materials are used, because proper linings and steel balls are unobtainable. Many of the kilns are in no better state. Germany has neither magnesite nor bauxite, and cannot therefore now make refractories of these materials. The kilns, often lined with low-grade firebrick and in many cases with clinker-concrete blocks even in the burning zone, can hardly be expected to produce high-grade clinker. The types of fuel which are essential for the maintenance of a proper burning process are seldom available. When it is considered that Portland cement clinker has had to be made in rotary kilns with brown coal (lignite), there will be no surprise that the product contains 2.5 per cent. and more of free lime. A proper judgment on cements used in the construction of the autobahnen is possible only by reference to the conditions when these cements were made, the German standard specifications, and to the special regulations imposed on the quality of the "autobahn cements."

German and American Cements.

A comparison of German and British standards for cement is difficult, but a comparison with the standards of the United States is almost impossible. Concep-

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tions as to what tests should be made and how they should be made differ greatly in different countries. Difficulties begin with the classification of cements. There are five types in America. The question of the desirability of classifying cements only in accordance with their strength, or in accordance with other characteristics, has been discussed in Germany for many years. The writer in 1934 suggested⁴ the establishment of four types of cement in addition to ordinary Portland cement, namely (1) Rapid-hardening cement described as "Reinforced-concrete cement"; (2) Cement with low shrinking properties, described as "Road-making Cement"; (3) Low-heat cement described as "Mass-concrete Cement"; and (4) Cement with good resistance to chemical action. It is interesting that these four types to a large extent correspond with those, additional to ordinary Portland cement, in the latest standards of the United States.

The reasons why the German cement industry has not adopted the writer's suggestions are probably that at that time less knowledge was available and that the war started a few years later. It was obviously impossible, during and since the war, to make such a radical alteration to the German standards. In general, cements were made in two (later three) qualities in accordance with their hardening properties and a special type of "Autobahn cements" was created. In this type the low-shrinking characteristics were most important.

Chemical Composition.

While the specifications for the chemical composition of cements in Germany, Britain, and most other countries were limited to restrictions on the magnesia content the sulphuric-acid content, the loss on ignition, and the insoluble residue, standards in the United States give very detailed instructions for the analysis of each of the five types of cement, and there are also standards for the main mineral components of the clinker: tricalcium-silicate, bicalcium-silicate, and tricalcium-aluminate. For some cements there are limitations of the heat developed during setting. On the other hand it appears possible that cements which do not satisfy the chemical requirements of the standards of the United States may also be quite satisfactory for these purposes. In none of the five cements in the United States is the iron-oxide content permitted to exceed 6.5 per cent., but in Germany a cement known as Erz-zement (iron-cement), which often has a far higher content of iron oxide, is not only suitable for building purposes but is also resistant to chemical attack. The writer is therefore of the opinion that a cement should not be condemned merely because it does not comply with one or other of the many specifications. Such cases are, however, exceptions, because most German cements made before the war would probably have satisfied all American regulations for chemical composition.

The United States standards, in addition to limiting the amount of the different oxides, restrict the content of the main mineral components (tricalcium-silicate, etc.). There is little reason to discuss this here, particularly as these limits are useful only in a mathematical sense insofar as the amounts of the mineral components depend on the speed of cooling and because they change with manufacturing conditions. It also appears that, in spite of the very excellent work of



Mr. L. A. Dahl, the formulæ suggested by him ⁵ may require alteration, particularly because the latest American research has drawn attention to the effect of alkali and because Mr. R. H. Bogue⁶ has drawn attention to the fact that this also affects the combination properties of the lime.

Fineness.

A comparison of the requirements of fineness is impossible because it is measured by specific surface in a turbidimeter in the United States, and by the residue on sieves in Germany. The writer does not accept the opinion expressed in the American report that American cements must in any case be finer because they are mostly ground in closed-circuit separator mills, while multiple-chamber mills are preferred in Germany. According to German experience the case is exactly the reverse. It has been proved that the strength of cement ground in compound mills, particularly the early strengths, is higher than that of cement ground in closed-circuit mills with air separators. This is ascribed to the fact that the "fines" are ground still finer in a compound mill while they are taken out of the grinding process as soon as they have attained a specific fineness in a closed-circuit mill.

Strength.

A comparison of the standards regarding strength is made difficult by the change-over in Germany to tests on plastic mortar with a relatively high water

TABLE I
Tensile Strength. 1 : 3 Mortar. Lb. per square inch.

Time	Country	Ordinary Portland cement Type I	Rapid-hardening cement	
			Type II	Type III
1 day	Germany (1942)	—	—	<u>356</u>
	Britain (1947)	—	300	—
	United States (1947)	—	275	—
3 days	Germany (1932)	—	356	—
	Germany (1939)	—	<u>356</u>	—
	Germany (1942)	—	<u>427</u>	<u>711</u>
	Britain (1947)	300	450	—
	United States (1947)	150	375	—
7 days	Germany (1932)	256	—	—
	Germany (1939)	<u>356</u>	—	—
	Germany (1942)	<u>356</u>	<u>569</u>	<u>853</u>
	Britain (1947)	375	—	—
	United States (1947)	275	—	—
28 days	Germany (1932)	356	427	—
	Germany (1939)	<u>711</u>	<u>783</u>	—
	Germany (1942)	<u>711</u>	<u>853</u>	<u>996</u>
	United States (1947)	350	—	—

Notes. 1. The figures underlined are bending strengths of plastic mortar with 15 per cent. water.

2. The tensile strengths specified in the British standards are based on direct tensile tests on briquettes.

JANUARY, 1949

CEMENT AND LIME MANUFACTURE

content. This made it necessary to use a special standard sand, the high content of fine grains of which may cause doubts. Nevertheless the change was made because it was justified by experience. A comparison of the strength of cement as prescribed in different countries must always take account of the method by which the strength is measured. For example, in Britain and the United States sands of almost identical grain size, between 0.6 mm. and 0.85 mm., are used, while in Germany two sands are used of which one (with grains of 0.74 mm. to 1.39 mm.) is much coarser and the other (with grains of 0.09 mm. to 0.2 mm.) is much finer than the former. Although tensile tests or bending tests are made in all three countries with 1 : 3 mortar test pieces there is a difference so far as compression tests are concerned; a 1 : 2.75-mortar is used in the United States. The difference in the water-content of test pieces in different countries is most important. British standards prescribe about 8 per cent. of water for tensile-test briquettes and about 10 per cent. for compression tests. In the United States the proportion of water is a little more, but they do not reach the proportion of 15 per cent. specified in Germany. It must further be noted that tests for bending-strength in Germany are made in accordance with new standards and that the results obtained are generally much higher than those obtained with tensile tests made in accordance with the former standards. Finally, the high water-content results in a lower strength, not so much at 28 days, but at earlier ages.

TABLE II
Compressive Strength. 1 : 3 (1 : 2.75) Mortar. Lb. per square inch.

Time	Country	Ordinary Portland cement. Type I	Rapid-hardening cement.	
			Type II	Type III
1 day	Germany (1942)	—	—	<u>1422</u>
	Britain (1947)	—	1600	—
	United States (1947)	—	1250	—
3 days	Germany (1932)	—	3556	—
	Germany (1939)	—	3556	—
	Germany (1942)	—	<u>2134</u>	<u>4267</u>
	Britain (1947)	1600	3500	—
	United States (1947)	900	2500	—
7 days	Germany (1932)	2560	—	—
	Germany (1939)	2845	—	—
	Germany (1942)	<u>1565</u>	<u>3200</u>	<u>5120</u>
	Britain (1947)	2500	—	—
	United States (1947)	1800	—	—
28 days	Germany (1932)	3910	5689	—
	Germany (1939)	4267	5689	—
	Germany (1942)	<u>3200</u>	<u>4623</u>	<u>6045</u>
	United States (1947)	3000	—	—

The figures underlined indicate the compressive strengths of plastic mortar with 15 per cent. water.

In Tables I and II are summarised the strengths (converted to British units) prescribed in Germany in 1932, 1939 and 1942,* and the requirements of the present standards in Britain and the United States.

When comparing the requirements for the hardening of cements by these standards it is seen that German cements are not inferior to others, particularly when the considerable differences in the methods of testing are taken into consideration.

Expansion and Shrinkage.

The importance of constancy of volume is appreciated in Germany as much as elsewhere. In America the danger of expansion appears to be considered more serious than that of shrinking; at least this may be inferred from the introduction in the United States of the autoclave test and the absence of any requirement regarding shrinkage. Opinion in Germany is the exact opposite, probably because German cements have not suffered from expansion for many years. In Germany it is thought that the hot-pat test or the Le Chatelier test provides sufficient safeguard against expansion due to lime and that expansion due to magnesia or gypsum is impossible if the prescribed maximum amounts of magnesia and SO_3 are not exceeded. For these reasons it is not believed in Germany that the autoclave test is necessary, and it is not without interest that the autoclave test was introduced in the United States only after considerable discussion, that it has not been introduced in Britain or in many other countries, and that recently serious doubts about the autoclave test have been expressed, for example by Mr. J. M. Balaguer⁷ in Barcelona and Mr. H. K. Vaughan in the United States. The latter writes: "Excessive expansion with the autoclave test in no way proves that the cement is of poor quality, but it does with the Le Chatelier test."

While experience in Germany has shown that damage to concrete structures as a result of expansion is extremely rare, the dangers arising from shrinking are very much greater. The writer believes that the shrinking characteristics of a cement are more important than its strength. Few concrete structures deteriorate because of insufficient strength of the cement, but many structures develop cracks and even small cracks are a source of danger because cracks not only allow chemicals to penetrate into the concrete but changes of temperature gradually increase the size of the crack.

The question whether dangerous cracks in concrete are really caused by shrinking or whether other forces, such as changes of temperature, play a decisive part, has been discussed in Germany in great detail. Dr. R. Grün⁹ and others have pointed out that there is no reliable similarity between small prisms and large concrete blocks so far as shrinking is concerned; it is recorded that shrinking tests with cubes proved satisfactory while concrete structures using the same cement were unsatisfactory, and vice versa. The writer does not agree, however, with those who think that shrinking of cements in general is of little importance in relation to the quality of concrete. It is only the external working conditions which are responsible for the differences in the shrinking effect of test cubes and

*A summary of German Standards for cement is given on p. 10 of this number.

JANUARY, 1949

CEMENT AND LIME MANUFACTURE

full-scale concrete structures, because shrinking is after all a natural characteristic of cement arising out of the contraction of the product due to the setting and hardening of cement and is not the result of differences between the sand used in a test specimen and the aggregate used in a concrete structure.

The opinion is sometimes held that the shrinking of cement cannot be of decisive importance in the development of cracks in roads because volume changes due to changes of temperature in road surfaces are considerably greater than those caused by the shrinking of the concrete on hardening. This view neglects the fact that volume changes resulting from thermic causes are reversible, while tension caused by shrinking remains and is increased as the concrete ages. This progressive contraction is a result of the colloidal character of set cement. There is a difference between reversible shrinking as illustrated by the case of gelatine, and non-reversible shrinking which leads to a rigid insoluble structure as illustrated by silica-gel. Cements belong to the group of non-reversible gels and the shrinking continues during the whole hardening period, and although the soaking of concrete in water leads to an increase in volume, the increase is not generally sufficient to regain the original volume. This is the basic difference between volumetric changes caused by shrinking and by thermic changes.

Such considerations do not, however, exclude the possibility of very varying effects of shrinking. A network of fine cracks on the surface of a concrete structure appears relatively harmless, and Dr. Graf¹⁰ has rightly pointed out that such hair-cracks are caused by over-rapid loss of water from the surface, that they do not penetrate into the heart of the concrete, and that they have little connection with those forces which lead to increasing tension in a large concrete structure and which finally lead to wide cracks through the concrete. It is this latter type of crack which must be considered responsible for damage to road surfaces, while hair-cracks will at most lead to slightly greater signs of wear and tear on the surface.

It might be thought that the formation of cracks due to shrinking might be avoided by thorough tamping or by vibration, whereby the concrete would be so dense that the particles of aggregate would touch each other and form a dense material which would resist contraction. Although such measures have been extensively used during the construction of the German motor-roads, and although they have some beneficial effect, it is clear that very close packing of aggregate can hardly prevent shrinking due to shrinking of the cement. The large tensile forces set up by this shrinking must somehow be relieved, and therefore lead to a contraction of the structure as a whole, or, if such contraction is restrained, to cracking.

On the other hand creep, as described by K. Walz¹¹, can lead to the relaxation of the tension, and if the concrete road slab can slide on its base, as was provided for in the German motor roads, the creep of the concrete can reduce the tensile force created by the shrinking of the cement, thus preventing the formation of cracks. This is thought to be the reason why cements containing blastfurnace slag have proved so satisfactory for these motor roads, since Dr. R. Grün⁹ has ascertained that an increase in the percentage of blastfurnace slag increases the tendency for the concrete to creep. It was such consideration which led to the estab-

lishment of special specifications for "autobahn cement." Details for these have been given by Dr. Graf¹² with particular reference to the preparation of the raw material and the burning process.

Experience had proved that the former testing methods using a low water-content in the mortar was unsatisfactory for the prediction of concrete strength, and had established that for such purposes mortar with a high water-content would have to be tested. The demand for a method of testing which would give better guidance regarding the behaviour of concrete in structures led to the introduction of a test-piece of plastic mortar with a water-cement ratio of 0.6 and to the adoption of bending instead of tensile-strength tests. This method of testing quickly proved so successful that it was also applied to compression strength tests, and is now always used in Germany.

In view of the lack of experience of the connection between the shrinking of cement and its other characteristics, and because it was realised that shrinking was of particular importance in concrete roads, attempts were made to meet the dangers resulting from shrinking by giving preference to cements with high bending strength and also by prescribing the chemical composition and fineness. The chemical requirements were based on the fact that shrinking is increased in proportion to the increase of alumina content. On the other hand the work of Dr. H. F. Gonnermann¹³ had shown that the shrinking of tricalcium-alumina is considerable while calcium-alumina-ferrite shrinks only a little. Because of this tendency of the most important minerals in the clinker, cements used for roads were chosen for their low alumina content and high ferric-oxide content. Greater fineness was desirable because of the greater strength, but this also meant increased shrinking. A compromise was agreed which specified a limit of 5 per cent. residue on sieve No. 0.09 (DIN 1171) which corresponds to British standard sieve No. 170.

The desirable characteristics of cement for roads was at that time determined by trial and error, because little research had been done in this direction. It was therefore not possible to draw up definite specifications for the cement for this purpose. There were guiding rules only, and they had to be supplemented by practical tests which were made with samples of cements from the works which were likely to be asked to supply the cement. These samples were subjected to comprehensive tests of all characteristics which were realised to be of importance. If they proved satisfactory, the works in question was asked to supply cement of identical quality and characteristics. The cement for use on the motor roads was stored apart from other cements, and tests were made to ensure that the road cement was in accordance with the original sample.

Test roads were built near Stuttgart, Cologne, Hanover and Berlin. These roads were built with cements of different chemical compositions but with identical constructional methods. They were closely examined regularly, and the results were published by F. Weise¹⁴, K. Eberle¹⁵, and O. Graf and K. Walz¹⁶. These reports agree on the importance of the "plasticity" of the cement during the preparation of the concrete. The concrete must be workable, but should not tend to separate. A good surface is assured with such cements but it is agreed

that the observations of only a few years do not allow one to draw any conclusions on the superiority of any one of the many cements used. Unfortunately the inspection of the roads was not continued fully during the war or since, and there are therefore considerable gaps in the experience which might have been gained.

This article has perhaps shown that very great care was taken with the selection of cements for the German motor roads, and that new methods were used for this purpose. It seems that the result has proved the suitability of the selected cements for the purpose for which they were intended. The question whether these cements satisfy the many requirements of the standards of the United States perhaps need not be answered; they have proved successful in practice and that is the important point.

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Determining the Fineness of Cement.

A METHOD that is not well known for the determination of the residue on a No. 170 sieve is to weigh out 10 grammes (accurate to 0.1 gramme) of the cement. This amount is spread on a small sieve having 170 meshes per linear inch and is washed for two minutes with a jet of water having a back-pressure of 10 lb. per square inch. The sieve (with the residue thereon) is then placed on a hot plate until all the moisture has evaporated; this may take four to five minutes. The dried residue is then tapped out on to the pan of a balance and weighed, and the weight is expressed as a percentage of 10 grammes. This method is quicker and cleaner than dry-sieving and, although not complying with British Standards, it is said to give results as accurate as the standard method.

The jet of water is sprayed on to the sieve through a nozzle formed from a $\frac{3}{4}$ -in. pipe-cap in which nine holes $\frac{1}{8}$ in. diameter are drilled; one hole is at the centre of the cap, and the others are equally spaced on a circle of $\frac{1}{4}$ -in. diameter.

German Specifications for Cement.

DURING the war revised specifications for Portland, iron-Portland, and blast-furnace-slag cements were published in Germany (DIN 1164, 1942). As copies of these specifications are not readily available in this country, we give in the following the principal requirements. For particulars of the revisions made in 1939 see this journal for December, 1939.

Three qualities of cement are recognised and are termed respectively No. 225, No. 325 and No. 425; the figures correspond to the compressive strengths of the mortar in kilogrammes per square centimetre at 28 days.

Portland cement must contain not more than 5 per cent. of MgO and not more than 3 per cent. of SO_3 .

Iron-Portland cement must contain at least 70 per cent. of Portland-cement clinker complying with the requirements for Portland cement and not more than 30 per cent. of quickly cooled blastfurnace slag. Blastfurnace cement may contain 15 per cent. to 69 per cent. of Portland-cement clinker and 85 per cent. to 31 per cent. of blastfurnace slag. The ratio of $(\text{CaO} + \text{MgO} + \text{Al}_2\text{O}_3)$ to SiO_2 must be not less than 1. The SO_3 -content of iron-Portland cement must not exceed 3 per cent., and of blastfurnace cement 4 per cent. Blastfurnace cement must contain less than 55 per cent. of CaO.

The loss on ignition of Portland cement must not exceed 5 per cent. Added foreign material, except gypsum and water, must not exceed 1 per cent. The residue on a sieve having 4900 meshes per square centimetre must not exceed 20 per cent. The hot-pat and cold-pat tests for soundness apply.

The tensile and compressive strengths are measured on specimens of mortar comprising 1 part of cement to 1 part of standard fine sand to 2 parts of standard coarse sand to 0.6 part of water. The specimens tested at three days or later are stored in water. The minimum tensile strengths in lb. per square inch as determined by a bending test are: Cement No. 225, 356 at 7 days and 711 at 28 days; cement No. 325, 427 at 3 days, 569 at 7 days, and 853 at 28 days; cement No. 425, 356 at 1 day, 711 at 3 days, 853 at 7 days, and 996 at 28 days. The compressive strengths in lb. per square inch are: Cement No. 225, 1565 at 7 days and 3200 at 28 days; cement No. 325, 2134 at 3 days, 3200 at 7 days, and 4623 at 28 days; cement No. 425, 1422 at 1 day, 4267 at 3 days, 5120 at 7 days, and 6045 at 28 days.

Production of Cement in Brazil.

THE Minister (Commercial) at Rio de Janeiro has reported that according to press information the production of cement in Brazil during the first half of the year 1948 amounted to 511,892 tons and that the total production during the year July 1, 1947, to June 30, 1948, was 913,525 tons.

Some New Ideas on the Composition of Cement.

IN the number of this journal for May 1948 the views of Mr. A. Rebuffat on hydrolitical reactions in cements were given, and in the following some of the opinions of Mr. Rebuffat on the composition of cement, and particularly on the part played by the silicates, are described. These ideas are embodied in a paper read by the author before the 20th Congress of Industrial Chemistry in Paris in 1946 and published subsequently in "Chemie et Industrie."

The author states that the composition of cement does not conform entirely with ideas hitherto held. For example, it appears that iron is combined with the silicates, that Brownmillerite ($4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Fe}_2\text{O}_3$) is found mainly in cements, such as slag cements, made with materials rich in aluminiferous compounds, and that zeolite (that is hydrated silicate of calcium and aluminium) is found in any hydrated cement (that is for each molecule of combined alumina there are three or more molecules of silica).

Analysis of the liquors of hydrolysis showed that, with Portland and pozzolana cements of 59 to 61 per cent. lime content not more than 15 per cent. of silica was present, which is much less than would be required if all the lime were combined as tricalcium silicate. The author does not doubt that there is also a substantial amount of tricalcium-silicate as indicated by the spectrograms of, among others, Guttman and Gille, but finds it impossible to suppose that the zeolite is formed by recombination of silica, alumina and lime set free from other materials by hydrolysis. For this various reasons are adduced. It seems to the author that the lime in these liquors originates from a very basic ternary compound which is perfectly stable in dry air but which in contact with water sets free lime, absorbs water, and swells. It is pointed out that the hypothesis of a solid solution of lime or calcium-aluminate in a calcium-silicate was definitely rejected at the Zurich Congress of 1931. By the methods described in the paper on hydrolysis it is possible to reconstruct the composition of the aluminate-silicate of lime producing the zeolite. It is sufficient to calculate the amount of lime, which is freed by hydrolysis and which can result from neither the tricalcium-silicate nor from the tricalcium-aluminate, and deduct the free lime in the clinker. It is also necessary to know whether the cement contains any SO_3 or calcium sulphate.

There are two methods of approach, namely, one from the amount of lime in the original clinker and the other from the amount of lime freed by hydrolysis; consequently there is a check to a certain degree. The results of calculations are given in *Tables 1* and *2*, wherein attempts to reconstitute the clinkers of three types of Italian cements are shown. The references II, III and IV relate to some of the clinkers described in *Table 1* on page 55 of our number for May, 1948. Clinker II (*Table 1*) is a Portland cement having an hydrolysis number 60.1, and aluminosilicate, $3\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot 11\text{CaO}$. Clinker No. III (*Table 1*) is also a Portland cement with an hydrolysis number 61.5, and aluminosilicate, $3\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot 9\text{CaO}$. Clinker No. IV (*Table 2*) is a pozzolana cement with an

hydrolysis number 61.0, and aluminosilicate, $4\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot (\text{Na}_2\text{K}_2)\text{O} \cdot 13\text{CaO} \cdot \text{MgO}$; the distribution of the alkalis and the magnesia cannot be determined precisely.

It is found that the compositions of the aluminosilicates differ with the type of clinker and that, for clinkers of the same type, if the zeolitic formation is the same the basicity of the aluminosilicate can differ. In samples of the same

TABLE 1.—PORTLAND-CEMENT CLINKERS.

	SiO_2	Al_2O_3	CaO	Fe_2O_3
Clinker No. II Analysis :	22.27	5.49	67.00	3.01
Free lime			0.38	
Tricalcium-silicate	11.55		32.26	
Aluminates (free ?)		0.25	0.39	
Celite	1.50		2.11	3.01
Alumino-silicate	8.80	5.00	30.25	
Quartz, etc.	0.85		1.50	
	22.70	5.25	66.89	3.01
Clinker No. III Analysis :	23.70	5.24	67.40	2.31
Free lime			0.73	
Tricalcium-silicate	14.23		39.84	
Aluminates (free ?)		0.24	0.39	
Celite	0.86		1.61	2.31
Alumino-silicate	8.80	5.00	24.75	
Quartz, etc.	+		+	
	23.89	5.24	67.32	2.31

TABLE 2.—POZZOLANA-CEMENT CLINKERS.

	SiO_2	Al_2O_3	CaO	Fe_2O_3	MgO	Alkali	SO_3
Clinker No. IV Analysis :	22.36	3.28	65.34	2.52	1.42	2.34	1.30
Free lime			—				
Tricalcium-silicate	13.47		37.52				
Aluminates (free ?)		0.00					
Celite	1.19		2.18	2.52	0.26		
Alumino-silicate	7.70	3.28	23.42		1.27	2.49	
Calcium-sulphate			1.07				1.30
	22.26	3.28	64.19	2.52	1.53	2.49	1.30

original composition, but subjected to different degrees of calcination, the composition of the aluminosilicate can vary with excessive calcination. It appears that the rate of decomposition of the aluminosilicate in the clinkers is directly proportional to the basicity. For example, in Portland cement No. II the proportion of CaO is 11 to aluminosilicate 3; in Portland cement No. III the proportion of CaO is 9 to aluminosilicate 3; in pozzolana cement No. IV the proportion of $\text{CaO} \cdot \text{MgO}$ is 13 to alkaline aluminosilicate 4. The rate of hydrolysis of cement No. II is greater than that of No. III, and the rate of setting is appreciably greater.

Another point of fundamental significance is that in samples of normal composition when properly burnt no evidence was found of the pre-existence of free $2\text{CaO} \cdot \text{SiO}_2$. So far it may be concluded that in average samples the composition is about 40 to 45 per cent. tricalcium-silicate, 35 to 45 per cent. aluminosilicate with some alkali and magnesia in pozzolana cement, 5 to 7 per cent. calcium-silico-ferrite, and small amounts of quartz and non-hydrolysable silicates in Portland cement. Some of the iron is present as Brownmillerite in slag cements.

It is easy to understand why two samples of the same or very similar composition may have very different rates of setting and hardening. The proper role of the tricalcium-silicate is that of a diluent, and a very useful one, since too much aluminosilicate would result in quick setting and, if bicalcium-silicate is used as a diluent, an inferior cement is obtained. Too much alumina would certainly cause expansion. The formation of zeolite appears to take place in two stages; in the first there is expulsion of lime and in the second absorption of water and expansion, although these two stages may often overlap. Tricalcium-silicate is also hydrated, resulting in the evolution of heat, but in about three hours it may have appreciably decomposed and will then react much more slowly, especially in the final stages. It may be supposed that the ratio of the content of tricalcium-silicate to that of aluminosilicate is a governing factor in the setting of cements.

In the formation of the clinker two points must be considered: first the relation between the raw materials and the final product; and secondly what the author calls the "recurrence" of the properties of the clinker. In Portland cement there is always a mixture of calcium-carbonate, aluminohydrosilicate, free silica (quartz or amorphous silica), and complex silicates of iron and other substances. The principal constituents are the aluminohydrosilicates in the clay. One of these, kaolinite ($2\text{SiO}_2 \cdot \text{Al}_2\text{O}_3 \cdot 2\text{H}_2\text{O}$), is considered. It is worth while examining the theory that upon heating kaolinite the product is metakaolin ($2\text{SiO}_2 \cdot \text{Al}_2\text{O}_3$). One starts with a ternary compound containing silica, alumina and the water of constitution by means of which the reactions take place. The reactions are complex but invariably are processes of substitution and addition whereby lime is eventually combined in some manner with the other constituents. The proof of the theory has always been there, the author states, without it being realised, possibly because certain fundamental research has remained almost unknown, for example, that in Italy in 1902 by O. Rebuffat. Despite the work of Pukall, O. Rebuffat, and others, showing among other things that kaolinite during dehydration acquires marked chemical activity, Sosman asserted in 1933 his belief in the formation of metakaolin, although there was no definite proof, and in 1934 Dorsch referred to the chaotic changes taking place.

Evidently there is a phenomenon, inexplicable by orthodox means, concerning which the author presents a new hypothesis. The ideas of Hoffmann and others relating to the definite compounds containing both silica and alumina need to be supplemented by assuming that there is a silica phase and an alumina phase. These two distinct phases are in close contiguity but to a certain extent are independent. The connecting link is the water of constitution, which is not outside the system as appears to have been assumed by Asch, Groth, Mellor, and others.

In other words the essential difference in the new theory is that the water molecule (or hydroxyl) replaces the oxygen atom as the link. There is apparently no difficulty in supposing that the silica and alumina are united by the bond $>OH-O-HO<$. In fact it is known that the method of dehydration of clays is much more significant than their composition. In any case the water may be driven off by heat when the systems probably approach each other but do not change their relative positions. These residues of dehydration may have properties of oxide mixtures as well as of bodies with marked chemical activity. The independence of the silica and alumina systems enables them to conform to the laws governing their allotropic changes; and it is known that they follow opposite paths for the silica decreases in density while the alumina tends invariably to polymerise. It is clear that, at a certain moment, with the temperature rising, there is in the crystalline system actual distortion, when the substance becomes a true mixture of oxides. If the temperature rises further there is a direct reaction between the silica and alumina, and the resulting compounds have special properties. The heating curve of kaolin shows clearly irreversible changes, and this fact may explain most of the phenomena observed.

It is not desirable to retain the name of metakaolin, and "silal" is suggested for a substance having at the same time the properties of an oxide mixture and of a complex acid. Aluminohydrosilicates can then be called simply hydrosilals with considerable gain in clarity. The conception of silal, which enables one to go more deeply into the difficult problem of the constitution of certain silicates, makes it necessary to study the works of Weyer [*Zement* (1931) 96, 264, 560] from which the implications are more fully appreciated. Heating mixtures of kaolin and lime exemplifies reaction between substances in a solid state. Weyer noted correctly that the lime diffuses into the pholerite crystals and attaches itself to their interior, but his conclusions do not appear to be correct. The explanation given by O. Rebuffat relates mainly to the remarkable increase in volume which occurs on the heating of clayey limestones the composition of which is such that a quick-setting cement is obtained on heating. This was observed for the first time with the limestones from Montferrat which gave, by simple calcination at 800 deg. C., a very good quick-setting cement. The characteristic swelling was also noted in the heating of lime-kaolin mixtures, for example of 1 mol. kaolin with 3 to 7 mol. of lime, but the cementitious quality is reduced or destroyed by burning at a higher temperature.

Attempts to reproduce from mixtures of silica, alumina, and lime the same products that are easily obtained by heating kaolin or clay with lime invariably failed, although the product may have had good expanding properties. Bied has noted the large number of calcium-alumino-silicates which occur naturally, so that the chances are much in favour of obtaining ternary rather than binary products in the kiln. It is true that many of these silicates fuse abnormally, but this is no reason why it should be impossible to reproduce them by sufficiently prolonged heating of suitable mixtures, as in the ternary compound $SiO_2 \cdot Al_2O_3 \cdot 3CaO$. It is necessary to get used to the idea that, with the silicates, the equilibrium diagrams represent limits only attained after complete fusion. Seeger has already

noted that the points of fusion of silica-alumina mixtures do not correspond to the Bowen-Greig diagram, possibly owing to impurities. O. Rebuffat, in his paper on the silica-alumina system (*Giorn. Chim. Ind. e Appl.* 1934, 16,433), suggested that work in this field should be renewed and extended, with particular reference to the rising-temperature phase and irreversible equilibria. He found that, although much of the quick-setting property of the resulting cement was lost when heated to 1,200 deg. C., it reappeared to some extent on prolonged heating. He supposed that lime saturation occurred in a mixture in which the ratio corresponded to bicalcium-silicate and bicalcium-aluminate. It is well to recall the observations of Weyer on lime diffusing into the silal, taking the place of water, and probably reaching saturation stage, in which case the product obtained would have the general formula $m\text{SiO}_2 \cdot n\text{Al}_2\text{O}_3 \cdot (2m + 3n)\text{CaO}$, that is $\text{S}_m\text{A}_n\text{C}_{2m+3n}$, since the more basic compounds that the silica and alumina may form with the lime are, at temperatures of 1,300 deg. C. or less, bicalcium-silicate and tricalcium-aluminate. Is this a true ternary compound or only a mixture? The answer is that we have now a molecular crystalline aggregate formed with complete regularity; but it is not a mixture, for O. Rebuffat's experiments show that, as already stated, a mixture of silicates and aluminates, however intimate, cannot have, even when heated, the properties of a product obtained from the kaolin-lime mixture. Nevertheless if the saturation formula is seemingly valid in some cases (for example, cement No. III) it must be acknowledged that still more basic ternary compounds may sometimes be formed (cement No. II), and some explanation may be attempted.

The diagram of Kühl and Lorentz, reproduced by Dorsch ("Chemie der Zemente," 1932), for burning clinker gives for each temperature the amount of limestone (CaCO_3), free lime, and combined lime. Dorsch remarks that the curves are the combined effects of superimposed reactions, but that there is always some coincidence in curves obtained with raw materials of different composition. It would seem that the reactions take place according to a definite process and by stages. The diagram is of value, but the percentage analyses of the authors cannot necessarily be accepted, and the same applies to Weyer's figures. It is necessary to correlate the hardening, the composition, and the degree of burning with the results obtained by O. Rebuffat and the recurrence of certain phenomena, for example, the formation of neutral products. In the diagram of Kühl and Lorentz the percentage composition of the resulting clinker was: silica, 22.23; lime, 67.62; alumina, 3.44; ferric oxide, 3.22; magnesia, 2.37; sulphur trioxide, 0.92. The diagram shows pauses, at about 900 deg., 1,100 deg., and 1,200 deg. C., in the increase in the amount of combined lime, and consequently in the decrease in the amount of free lime. If reaction occurs at these temperatures it must be such that the total amount of free and combined lime does not change.

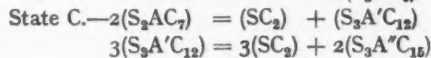
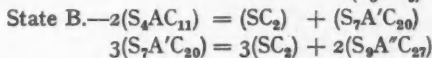
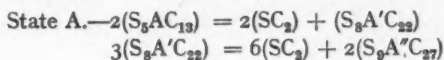
Quick-setting clinkers obtained by burning calcareous clays or lime-kaolin

mixtures at 700 deg. to 800 deg. C. deteriorate if they are heated above that temperature; the volume is reduced and the rate of setting is slower, and it may be supposed that the alumina is polymerised and that the whole change is gradual. References to the literature do not reveal any complete coincidence when different raw materials or degrees of heating are used. Mellor and Holdcroft found that the exothermic reaction took place at 800 deg. C., the density increasing from 2.82 at 600 deg. C. to 3.92 at 1,200 deg. C. Le Chatelier observed from thermal analysis that the form of the curves depends on the original oxide used. Houldsworth and Cobb note exothermic reaction between 1,060 deg. C. and 1,130 deg. C. A. B. Searle states that when heated to high temperatures bauxites polymerise and yield inferior results. The clay-orthoclase mixtures increase in density at 1,100 deg. C. and more at higher temperatures. This happens also with mixtures of clay and mica. The possibility of polymerisation is suggested in Pukall's experiments. It is not therefore unreasonable to suppose that the pauses in Kühl and Lorentz's curve show that condensation has occurred.

Besides the simple percentage composition of oxides, the mineralogical constitution should also be taken into account. Kühl and Lorentz's diagram shows that at 800 deg. C. 13 per cent. of the lime is already combined, and Weyer has noted that there is absorption of lime even at 400 deg. C. At 900 deg. C. it may be assumed that the silals are already saturated with lime and, assuming that all the alumina is combined in the silals, the percentage of combined lime shown by the curve is still higher than would be required for simple saturation; further, if there is sufficient alumina in the original charge a quick-setting product is obtained. Spangenberg (*Zement*, 1931, 94) supposed that at 860 deg. C. there may be some alumina in the silica system, judging from the density of the metakaolin and the exothermic reaction. It may be assumed that two molecules of the crystalline aggregate formed by saturation of the silal condense and expel one or more molecules of bicalcium-silicate. This would correspond to the pause at about 900 deg. C. on Kühl and Lorentz's diagram and explain the phenomena observed; this is the silicate noted by Weyer in his refraction index.

The author's discussion of the diagram also relates to the slope of the curve immediately after the pause at 900 deg. C., and the various forms of silica present, with the possible conversion of tricalcium-silicate into the bicalcium form at the pause at about 1,100 deg. C. This stage is followed by the pause at 1,200 deg. C. corresponding to a second polymerisation. At 1,250 deg. C. the clinker still contains much bicalcium-silicate, an objectionable rather than a useful diluent as it tends to pulverise quartz, sesquioxide of iron, and free lime. The quality of the product is directly related to the composition of the original charge. With sufficient clay to start with there will be a preponderance of alumino-silicate and a quick-setting product.

Assuming that saturation is indicated by the formula $S_m A_n C_{2m+3n}$, and that condensation produces Al_2O_3 , as suggested by W. and D. Asch, various states of polymerisation may be represented as in the following, in which A denotes Al_2O_3 , A' denotes Al_4O_6 , and A'' denotes Al_6O_9 .



States A and B may apply to cement No. III. It is less easy to determine the formula for the aluminosilicate of cement No. II and there is no proof that these states are correct. State C seems to correspond well to white kaolin cement which, according to Fritsch ("Fabrication du Ciment," 1920) is a true Portland cement, thus differing from some white cements.

Reverting again to Kühl and Lorentz's diagram, at about 1,250 deg. C. the presence of iron begins to be shown and the colour changes. In a rotary kiln this is a somewhat difficult stage. Free lime is now about 18 per cent., but if the original charge contains little quartz and much combined silica this percentage may be lower. The formation of bicalcium-ferrite is bound to occur, although there is a possibility that under certain conditions metaferrite may form, and this fuses at about 1,216 deg. C.

Reference is made to the work of B. Tavasci on the systems lime-ferric oxide and lime-alumina-ferric oxide (*Ann. Chim. Applicata*, No. 7, 1936, and No. 2, 1937). Tavasci produced some samples by simple burning at 1180 to 1190 deg. C., but his diagrams, like others, are only valid if one really starts with mixtures of oxides or if these are formed by burning. If this is not so equilibrium, but of varying kinds, may be attained temporarily. Nevertheless these experiments confirm the role of mineraliser attributed by Hägermann to iron (*Tonind. Zt.*, 1932, 28). The silico-ferrite of lime surrounds the lime still free and surrounds it with the bicalcium silicate, which, if there is sufficient lime, is thus completely transformed into crystalline tricalcium silicate. In order to effect this without any flux an appreciable amount of lime would be required, and the author agrees with J. Hendrick that crystallisation of the silicate is necessary.

At 1450 deg. C. burning may be considered complete for Portland cement, and further rise in temperature does not improve the product but, on the contrary, may worsen it. The aluminosilicate is generally only a temporary product which, stable at ordinary temperatures, tends to decompose when heated, gradually changing into bicalcium silicate and a residue which may be a stable aluminosilicate. Over-burned Portland cement clinkers contain bicalcium silicate with iron distributed throughout the structure of the clinker and have a glassy appearance. O. Rebuffat and V. Mazetti (*Atti R. Inst. Incoraggi*, Napoli, 1930) showed

that over-burned clinker when slowly cooled fell to powder from which, by simple sifting, prismatic micro-crystals of a composition corresponding to $4(\text{SiO}_2 \cdot 2\text{CaO}) + \text{Al}_2\text{O}_3 \cdot 3\text{CaO}$ were also obtained. It should be possible with suitable materials to produce natural aluminosilicates under atmospheric pressure and sufficiently prolonged burning at a definite temperature.

Although two cements or clinkers may have practically identical compositions they may differ in quality due to the amount and nature of the calcium aluminosilicate, the amount of tricalcium-silicate, and the presence of bicalcium-silicate. It is essential to have precise knowledge of the raw materials used.

The author makes no reference to the petrography of clinkers, partly because he thinks it is a serious error to assume that only a polarising microscope can solve the problem of the composition of cement. Some difficulties and doubts in this method of examining clinker are discussed, including the absence of any considerable literature and the possibility of unreliable results. No reference is made to Bragg's X-ray studies of silicate structure. New structure models are doubtless required, and work on the lines of that of Weyer might well be continued and extended, including further examination of belite and epeizite.

The existence of a very basic aluminosilicate capable of reacting with water has suggested the idea that, in the formation of natural zeolites through hydrothermic action, one may postulate the pre-existence of a very basic aluminosilicate of probably magmatic origin followed by the action of water. This view is said to be supported by geological evidence. The foregoing ideas are largely theoretical, and further study and experiment are required.

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	1939	End of 1947
Transport of coal ..	244	269
Transport of cement ..	268	311
Price of coal ..	140	408
Labour costs ..	243	430
Price of cement ..	111	172

The price of cement (in Swiss francs per ton) in Switzerland and in some other countries are : Belgium, 61.56 ; Switzerland, 64.6 ; Britain, 65.35 ; Germany, 70.70 ; France, 72.5 ; and Austria 97.10.

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